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# *rac*-(1*R*,11*S*,12*S*)-12-Iodo-15-oxobicyclo[9.3.1]-pentadecane-1-carboxylic acid ethyl ester

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**Key indicators**

Single-crystal X-ray study
$T$ = 291 K
Mean $\sigma$(C–C) = 0.007 Å
$R$ factor = 0.040
$wR$ factor = 0.107
Data-to-parameter ratio = 17.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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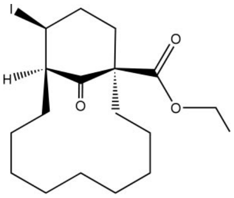
The title compound, $C_{18}H_{29}IO_3$, features a *trans*-fused bicyclic system. The carbonyl group lies on the same side as the carbethoxy function and iodine. The six-membered ring has a chair conformation.

## Comment

In order to obtain a 14-membered ring, the title compound, (I), was prepared *via* the Appel reaction from the corresponding alcohol, and subsequently was used as the starting material in a study of the application of the Grob fragmentation. Conversion of *rac*-(1*R*,11*S*,12*R*)-12-hydroxy-15-oxo-bicyclo-[9.3.1]pentadecan-1-carboxylic acid ethyl ester with $I_2$ and $PPh_3$ according to the previously reported general procedure (Lange & Gottardo, 2000) occurs *via* a nucleophilic substitution and, according to the reaction mechanism, the results involve an inversion of the configuration at C12.



(I)

## Experimental

To dry dichloromethane were added, in the following order, triphenylphosphine (1.5 equivalent), imidazole (1.5 equivalent) and iodine (1.5 equivalent). A solution of 2-hydroxy-15-oxo-bicyclo[9.3.1]pentadecan-1-carboxylic acid ethyl ester (1 equivalent) in dry dichloromethane was added and the mixture was stirred at room temperature under argon for 18 h. After completion of the reaction most of the solvent was removed under vacuum and the product was isolated by passing it through a column of silica gel with pentane as eluant, followed by three washings. Combining the fractions and evaporation of the solvent gave, without further purification, the pure crystalline product in 85% yield. The purity of (I) was confirmed by elemental analysis (calculated: C 51.43, H 6.95%; found: C 51.5, H 6.9%), NMR, IR and mass spectrometry: $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 4.62 (*m*, 1H, CH), 4.20 (*q*, 2H, $^3J$ = 6.4 Hz, $CH_2$), 2.90 (*td*, 2H, $^3J$ = 12.9 Hz, $^3J$ = 4.4 Hz, $CH_2$), 2.70 (*m*, 2H, $CH_2$), 2.36 (*m*, 4H, $CH_2$), 2.16 (*m*, 1H, $CH_2$), 1.40 (*m*, 1H, $CH_2$), 1.25 (*m*, 10H, CH), 1.23 (*t*, 3H, $^3J$ = 6.4 Hz, $CH_3$), 0.985 (*m*, 3H, $CH_2$), $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 206.13 (CO), 172.24 (CO), 61.95 ($C_q$), 61.19 ($CH_2$), 49.77 (CH), 39.88 (CH), 34.58 (CH), 33.13 ($CH_2$), 32.23 ($CH_2$), 30.57 ($CH_2$), 26.89 ($CH_2$), 26.08 ($CH_2$), 23.90 ($CH_2$), 22.95 ($CH_2$), 22.78 ($CH_2$), 21.66 ($CH_2$), 20.88 ($CH_2$), 14.33 ($CH_3$), MS LR (FAB, 70 eV): $m/z$ (%) = 421.23 ($M^+$ + H), 2.78), 293 (100), 247 (50.39), 219 (45.70), 109 (23.36), 95 (44.94), 81 (37.65), 67 (47.10), 55 (42.79), 41 (55.97), 29 (45.11), high resolution mass (FAB, 70 eV): calculated 420.1161, found 421.1270, IR (KBr),$\nu$ [cm$^{-1}$] = 2927 (*s*), 2867 (*s*), 1745 (*s*), 1711 (*s*), 1468 (*m*), 1437 (*m*), 1366 (*m*), 1279 (*m*), 1208 (*m*).

*Crystal data*

$C_{18}H_{29}IO_3$
$M_r$ = 420.31
Monoclinic, $P2_1/c$
$a$ = 10.9460 (2) Å
$b$ = 15.0522 (3) Å
$c$ = 12.8224 (3) Å
$\beta$ = 114.4262 (8)°
$V$ = 1923.54 (7) Å$^3$
$Z$ = 4
$D_x$ = 1.451 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 13534 reflections
$\theta$ = 3.2–25.4°
$\mu$ = 1.68 mm$^{-1}$
$T$ = 291 (1) K
Plate, colourless
0.50 × 0.22 × 0.08 mm

*Data collection*

Nonius KappaCCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min}$ = 0.488, $T_{\max}$ = 0.878
13534 measured reflections
3506 independent reflections
2004 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.052
$\theta_{\max}$ = 25.4°
$h = -13 \rightarrow 13$
$k = -18 \rightarrow 18$
$l = -15 \rightarrow 14$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
$S$ = 0.90
3506 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.80 e Å$^{-3}$
$\Delta\rho_{\min}$ = −0.76 e Å$^{-3}$
Extinction correction: *SHELXL*
Extinction coefficient: 0.0029 (5)

H atoms were placed in calculated positions, with C—H = 0.96–0.98 Å, and were refined as riding, with $U_{\rm iso} = 1.5U_{\rm eq}$(C) for the methyl group and $1.2U_{\rm eq}$(C) for the others; the methyl groups were allowed to rotate but not to tip.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97, *PARST*95 (Nardelli, 1995) and *PLATON* (Spek, 2001).

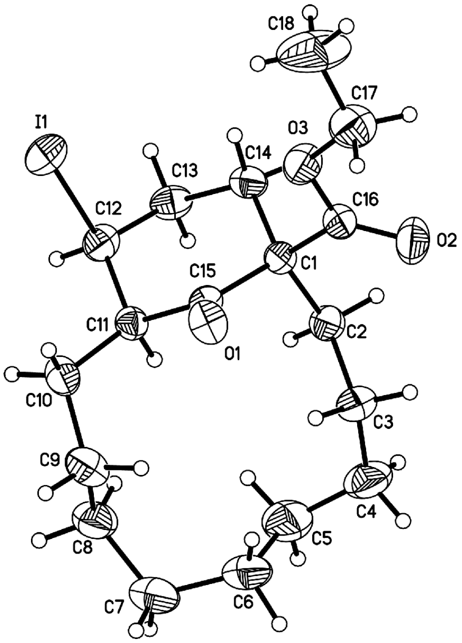


**Figure 1**
View of the title compound (*XP* in *SHELXTL-Plus*; Sheldrick, 1991), showing the labelling of all non-H atoms. Displacement ellipsoids are drawn at the 50% probability level.

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